### Remarks

In the Office Action mailed July 12, 2004, the Examiner maintained the rejection of claim 7 under 35 U.S.C. § 112, first paragraph.

The Examiner also maintained the rejection of claims 1-9 under 35 U.S.C. § 112, second paragraph.

The Examiner entered a new rejection of claims 1 and 5-8 under 35 U.S.C. § 102(a) or 102(f).

The Examiner also asserted a new rejection of claims 2 and 3 under 35 U.S.C. § 102(a) or 102(f).

In addition, the Examiner presented a new rejection of claim 9 under 35 U.S.C. § 103(a).

In view of the explanations set forth below, it is submitted that all of claims 1-9 are in condition for allowance.

Before turning attention to the Examiner's rejections, it is instructive to reconsider the subject matter of the present claims. The claims at issue recite a coating composition which provides corrosion resistance by the use of zinc alloy in flake form having particular proportions of zinc and other non-zinc alloy metals. It is noted that it is known to use mixtures of zinc particles and non-zinc metal particles in coatings. However, the claims at issue are all directed to corrosion resistant coatings containing zinc alloys, and particularly, zinc alloys in flake form having certain proportions of zinc and other non-zinc alloy metals such as for instance aluminum or tin. It is the use of these particular zinc alloys in a coating composition for providing corrosion resistance, and the particular coating compositions themselves, that represents a major advance in this field of art.

### A. Improper to Designate Office Action of July 12, 2004 as Final

The Examiner designated the July 12 Action as final. That designation is improper and premature since several grounds of rejection were raised in that Action for the very first time. In addition, the Examiner cited art against the pending claims for the very first time, i.e. U.S. Patent 4,356,036 to Kaliardos.

The Examiner is respectfully reminded of the provisions from the MPEP which state:

The applicant who is seeking to define his or her invention in claims that will give him or her the patent protection to which he or she is justly entitled should receive the cooperation of the examiner to that end, and not be prematurely cut off in the prosecution of his or her application.

The examiner should never lose sight of the fact that in every case the applicant is entitled to a full and fair hearing, and that a clear issue between applicant and examiner should be developed, if possible, before appeal.

### Section 706.07 of the MPEP.

It is inequitable and simply unfair to designate the July 12 Action as final. It is respectfully requested that the final designation be withdrawn.

### B. Rejection of Claim 7 Under § 112, First Paragraph, Must Be Withdrawn

The Examiner maintained the rejection of claim 7 under § 112 as follows:

Claim 7 is rejected under 35 U.S.C. 112, first paragraph, as failing to comply with the written description requirement. The claim(s) contains subject matter which was not described in the specification in such a way as to reasonably convey to one skilled in the relevant art that the inventor(s), at the time the application was filed, had possession of the claimed invention. Regarding Claim 7, it is unclear where the claimed alloy flake, having an unspecified amount of Zn as claimed, is supported by the original disclosure. Please see reasons of record in the Office Action mailed on 14 January 2004.

Page 2 from Office Action mailed July 12, 2004.

Claim 7 recites, in part, that the coating composition comprises "zinc alloy in flake form comprising a paste containing from about 4 to about 5 weight percent of aluminum in said alloy flake, and from about 7 to about 10 weight percent of paste liquid..." This preferred zinc alloy paste is described in the present application on pages 7 to 8. There, it is stated that:

The preferred alloy paste...is a paste of zinc and aluminum alloy in flake form typically containing from about 85 to about 86 weight percent zinc, from about 4 to about 8 weight percent aluminum and a balance of from about 7 to about 10 weight percent paste liquid, all basis 100 weight percent of the paste. Such a paste containing from about 4 to about 5 weight percent of aluminum in the alloy is also of particular interest.

Accordingly, the written description requirement is satisfied. The application as originally filed provides written description support for claim 7.

The Examiner asserted that it is unclear where the claimed alloy flake is supported by the disclosure. The previously quoted passage from pages 7 to 8 of the originally filed application provides the necessary support for the claimed alloy flake. Furthermore, the application as originally filed contains additional passages that provide support for claim 7.

The Examiner referred to the "reasons of record" in the Office Action mailed January 14, 2004. In that Action, the Examiner stated (regarding claim 7) that:

it is unclear where the claimed alloy flake, having an unspecified amount of zinc in a paste that contains 4-5 percent aluminum and 7-10 percent paste liquid, is taught.

Page 2 of the January 14, 2004 Action.

Support for claim 7 is found throughout the application. It is simply not understood why the Examiner is attempting to reject claim 7 for alleged lack of written description support under § 112, when in fact such support is present. For further support in the application as originally filed, the Examiner is respectfully requested to review page 7, lines 6-12; and page 35, lines 12-14. In addition, the Examiner is asked to note claims 5 and 7 as originally filed.

It is submitted that the Examiner will recognize that the present rejection under § 112, first paragraph, should be withdrawn.

### C. Rejection of Claims 1-9 under § 112, Second Paragraph, Must Be Withdrawn

In support of the rejection of claims 1-9 under § 112, second paragraph, the Examiner asserted:

Claims 1-9 are rejected under 35 U.S.C. 112, second paragraph, as being indefinite for failing to particularly point out and distinctly claim the subject matter which applicant regards as the invention.

I. Regarding Claims 1, 2, 4, 6, 7, and 8, it is unclear what is being admitted as the composition of the prior art. Applicant's explanations in the Interview Summary of 9 July 2004 and in the Response do not comport with the claim language that makes reference to elements in addition to a coating composition comprising liquid medium and particulate metal.

II. Regarding Claims 1, 2, 4, 6, 7, and 8, it is unclear what is required by the references to "curing" and "cured". Their plain meaning of the language suggests the presence of curing agent in the composition, but applicant has explained that no curing agents are required.

Pages 2-3 of the Office Action mailed July 12, 2004.

Concerning claims 1, 2, 4, 6, 7, and 8, it is not understood what the Examiner means by the statement "what is being admitted as the composition of the prior art." Those claims do not contain any admission as to prior art. Each claim is believed to recite patentable subject matter. The Examiner contended that these claims "make reference to elements in addition to a coating composition comprising liquid medium and particulate metal."

There is nothing in any of these claims which make reference to "elements in addition to a coating composition." For example, claim 1 recites, in part:

In a coating composition...which contains particulate metal in a liquid medium...the particulate metal comprising zinc alloy in flake form...

The Examiner is respectfully requested to re-review these claims. They are clear and definite.

Regarding claims 1, 2, 4, 6, 7, and 8, Applicant again notes that the claimed subject matter does not require the use of a curing agent. The term "cured" or "curing" refers to a chemical or physical process that improves the properties of the material undergoing curing. Specifically, in the claimed subject matter, curing refers to reactions, generally irreversible, occurring as a result of heating that improve the properties of the coating. No curing agent is required.

It is respectfully submitted that this ground of rejection be withdrawn.

### D. Rejection of Claims 1 and 5-8 under § 102(a or f) Must Be Withdrawn

In support of the rejection of claims 1 and 5-8 under § 102(a or f), the Examiner asserted:

Claims 1 and 5-8 are rejected under 35 U.S.C. 102(a or f) as being anticipated by Applicant's Admissions. Applicant's specification refers to prior art paste composition Eckart STAPA 4ZnAI7 zinc/aluminum alloy paste of the claimed alloy composition and liquid composition. See Specification (Example 1 – page 35).

Page 3 of the Office Action mailed July 12, 2004.

As evidenced by the enclosed Declaration, the Eckart STAPA paste noted in the present application is <u>not</u> prior art to the pending claims.

The Eckart alloy paste cannot be used as a prior art reference because the Eckart alloy paste derived from Applicant's own prior work.

Absent a statutory basis, an inventor's own prior original work will not anticipate his later inventions. In *In re Katz*, 215 USPQ 14 (CCPA 1982), the court stated:

Thus, one's own work is not prior art under § 102(a) even though it has been disclosed to the public in a manner or form which otherwise would fall under § 102(a).

See also Illinois Tool Works, Inc. v. Solo cup Co., 461 F.2d 265, 172 USPQ 385 (7th Cir. 1972); In re Faucius, 408 F.2d 1396, 161 USPQ 294 (CCPA 1969) ("But certainly one's own invention, whatever the form of disclosure to the public, may not be prior art against oneself, absent a statutory bar.").

As explained and evidenced in the enclosed Declaration by two of the named inventors of the present application, the STAPA paste available from Eckart was conceived and developed by the inventors themselves. That is, the Eckart STAPA 4ZnAl7 zinc/aluminum alloy paste was supplied to the assignee of the present application after development by the present inventors, and based upon information and specifications provided to Eckart by the inventors and/or their assignee. The noted STAPA paste resulted from the present inventors' own work.

In addition, the present rejection must be withdrawn because the cited pastes do not anticipate the claims at issue.¹ Claims 1 and 5-8 are all directed to a coating composition adapted for application to, and curing on, a substrate, in which the coating composition contains particulate metal in a liquid medium. The claimed coating compositions provide corrosion resistance as a cured coating on the substrate. The pastes cited by the Examiner do not anticipate claims 1 and 5-8. The pastes are not adapted for application to, and curing on, a substrate. The pastes do not provide corrosion resistance as a cured coating on the substrate. The pastes are not coatings. The pastes do not provide corrosion resistance as a coating. The paste is a component in the claimed

<sup>&</sup>lt;sup>1</sup> The Examiner is respectfully reminded that "[a]nticipation under Section 102 can be found only if a reference shows exactly what is claimed." *Titanium Metals Corp. v. Banner*, 778 F.2d 775, 227 USPQ 773 (Fed. Cir. 1985).

<u>coatings.</u> Simply put, the pastes fail to anticipate the corrosion resistant coating compositions recited in the pending claims.

Accordingly, the present ground of rejection must be withdrawn.

### E. Rejection of Claims 2 and 3 under § 102 (a or f) Must Be Withdrawn

The Examiner maintained the rejection of claims 2 and 3 under § 102 (a or f) for the following reasons:

Claims 2 and 3 are rejected under 35 U.S.C. 102(a or f) as being anticipated by Applicant's Admissions. Applicant's specification refers to prior art paste composition Eckart STAPA 4ZnSn30 zinc/tin alloy paste of the claimed alloy composition and liquid composition. See Specification (Example 2 – pages 37 and 38).

Pages 3-4 of the Office Action mailed July 12, 2004.

As explained and evidenced in the enclosed Declaration by two of the named inventors of the present application, the STAPA paste available from Eckart was conceived and developed by the inventors themselves. That is, the Eckart STAPA 4ZnSn30 zinc/tin alloy paste was supplied to the assignee of the present application after development by the present inventors, and based upon information and specifications provided to Eckart by the inventors and/or their assignee. The noted STAPA paste resulted from the present inventors' own work.

For the same reasons as previously expressed, the cited pastes do not anticipate the claimed coating compositions called for in claims 2 and 3. Those claims also recite a coating composition that is adapted for application to, and curing on, a substrate. And, those claims recite the composition as containing particulate metal in a liquid medium which provides corrosion resistance as a cured coating on the substrate. Simply put, the cited pastes do not anticipate the subject matter of claims 2 and 3.

In view of the enclosed Declaration and for the reasons expressed in the preceding section, this ground of rejection must be withdrawn.

### F. Rejection of Claim 9 under § 103(a) Must Be Withdrawn

In support of this ground of rejection, the Examiner argued:

Claim 9 is rejected under 35 U.S.C. 103(a) as being unpatentable over Kaliardos USP 4,356,036 in view of Applicant's admissions. Kaliardos teaches a zinc containing flake containing

paste, which further comprises non-alloy metallic particulate and other ingredients in order to provide a corrosion resistant metal coating material. See Kaliardos (Abstract; col. 4, line 41 through col. 6. line 44; and col. 7, line 45 through col. 8, line 61). Kaliardos does not exemplify a zinc/aluminum alloy paste as claimed. Kaliardos teaches that zinc containing flake paste in Kaliardos's compositions are effective to confer corrosion resistance. Applicant's Admissions teach a zinc/aluminum alloy flake paste of the claimed composition and structure that has corrosion resistance properties. See Specification (Example 1 page 35). In view of Kaliardos's teachings and suggestions, it would have been obvious to one of ordinary skill in the art at the time of the invention to fabricate the composition of Kaliardos with any effective zinc containing flake paste that provides corrosion resistance properties, including the zinc/aluminum alloy flake paste of Applicant's Admissions.

Pages 4-5 of the Office Action mailed July 12, 2004.

In view of the enclosed Declaration and for the previously noted reasons, the noted STAPA pastes are not prior art. The '036 patent to Kaliardos does not sufficiently teach or even suggest the subject matter of claim 9, and so, this ground of rejection must be withdrawn.

### G. Conclusion

It is respectfully urged that all pending claims, i.e. claims 1-9, are in condition for allowance.

Respectfully submitted,

FAY, SHARPE, FAGAN, MINNICH & MCKEE\_LLP

Richard J. Minnich, Reg. No. 24,175

Mark(E. Bandy, Reg. No. 35,788 1100 Superior Avenue, Suite 700

Cleveland, Ohio 44114-2579

(216) 861-5582

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### IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of : Maze

For : Particulate Metal Alloy Coating for

**Providing Corrosion Protection** 

Serial No. : 10/045,054

Filed : January 15, 2002

Art Unit No. : 1775

Examiner : Michael E. LaVilla

Date of Last Action : July 12, 2004

Confirmation No. : 2937

Attorney Docket No. : 003744-02

(MCII 2 00004)

Cleveland, Ohio 44114-2579

October 11, 2004

### **Declaration Under 37 CFR § 1.132**

We, Terry E. Dorsett, and Donald J. Guhde, are named co-inventors in the above-captioned patent application entitled "Particulate Metal Alloy Coating for Providing Corrosion Protection."

### We declare as follows:

- 1. We are co-inventors in the above-captioned patent application, along with Etienne Maze, Gilbert Lelong, and Toshio Nishikawa.
- 2. We are both employed by the assignee of the above-captioned application, Metal Coatings International, Inc. (MCII).

- 3. As we understand, the Patent Examiner in the above-captioned patent application rejected claims for corrosion resistant coating compositions based upon a paste composition designated as Eckart STAPA 4ZnAl7 zinc/aluminum alloy paste. This paste composition was noted (for example, on pages 5 and 35) in the above-captioned application filed on January 15, 2002.
- 4. As we understand, the Patent Examiner also rejected claims for coating compositions in that application based upon a paste composition designated as Eckart STAPA 4ZnSn30 zinc/tin alloy paste. This paste composition was noted (for example, on page 38) in the above-captioned application.
- 5. As we understand, the above-captioned application claims priority from a U.S. provisional application Serial No. 60/268,273 filed on February 14, 2001. The Eckart pastes were also noted in this provisional application.
- 6. As explained and evidenced herein, the Eckart pastes are thick, high viscosity pastes containing metallic alloys in flake or particle form. The Eckart pastes at issue originated from the work of the named inventors of the above-captioned application, i.e. Etienne Maze, Gilbert Lelong, Terry Dorsett, Donald Guhde, and Toshio Nishikawa, and specifically, originated from our work occurring prior to the filing date of the provisional application referred to in paragraph 5, i.e. February 14, 2001.
- 7. The first named inventor of the present application, Etienne Maze, is employed by Dacral. Dacral is a subsidiary of MCII.
- 8. At least as early as 1999, we, along with our co-inventors, identified several specific zinc alloys for use in the corrosion resistant coating compositions claimed in the above-captioned patent application. These zinc alloys included (i) a zinc/aluminum alloy containing 7% aluminum, and (ii) a zinc/tin alloy containing 30% tin.

- 9. It is believed that at the time period noted in paragraph 8, neither of the zinc alloys or those zinc alloys in a paste form, were commercially available.
- 10. In 1999, Etienne Maze, contacted Doral Corporation, a division of Eckart, to inquire as to the potential for Doral or Eckart to supply certain zinc alloys based upon specifications provided by Etienne Maze or Dacral.
- 11. It is believed that Doral forwarded the request and information of Etienne Maze to Eckart.
- 12. Personnel from Eckart visited Dacral in 1999. During that visit, Dacral requested Eckart to provide samples of zinc alloy flakes. These alloys included zinc alloys of tin, nickel, and aluminum. This visit and request is evidenced in item 4 of the trip report dated November 25, 1999 (see Exhibit A).
- 13. Eckart confirmed that it had produced a requested zinc alloy pigment, in a letter to Dorken AG (see exhibit B). In that letter the 90% zinc and 10% aluminum alloy pigment which Eckart had produced in response to the request from Dacral, is noted as "VP Zn 624 083." The "VP" designation refers to the product as an experimental product.
- 14. Nippon Dacro Shamrock Co., Ltd. (NDS) and MCII are subsidiaries of NOF Corporation.
- 15. MCII and NDS routinely work together in many different product developments. Often, NDS will conduct testing for or under the direction of MCII.
- 16. In early 2000, NDS was performing certain testing in conjunction with product development efforts by MCII. As evidenced in a correspondence dated February 7, 2000 from Kazuaki Sugano of NDS to Don Guhde of MCII (attached herewith as Exhibit C), NDS conducted various corrosion resistance tests. One test involved the use of an alloy paste nicknamed "4ZnAI7." See Attachment 1 to Exhibit C.

- 17. In response to the ongoing corrosion tests and the need for a supplier of various alloy pastes, Eckart was contacted to provide, among other materials, the paste nicknamed as "4ZnAl7." This paste corresponds to the zinc/aluminum alloy containing 7% aluminum referred to in paragraph 8. In addition, Eckart was requested to provide a zinc/tin alloy paste containing 30% tin, and nicknamed as "4ZnSn30." This paste corresponds to the zinc/tin alloy containing 30% tin referred to in paragraph 8.
- 18. On March 8, 2000, a letter was sent to Dean Fuller, Purchasing Manager for Metal Coatings, from Frank Passen of Eckart (see Exhibit D) confirming recent transactions involving various zinc pastes. In this letter, it is noted that a sample of the paste (referred to in this letter as ZnAl7) was delivered to Terry Dorsett of MCII.
- 19. Following the confirmation letter referred to in paragraph 18, Frank Passen of Eckart sent an email dated September 5, 2000 (see Exhibit E) detailing how Eckart would invoice MCII, NDS, and Dacral.
- 20. In late 2000, MCII conducted various testing involving the 4ZnAl7 paste. Specifically, within the period of December 19, 2000 to January 19, 2001, Janet French of MCII performed various tests using this paste. A copy of her laboratory notebook page 9924-2 is attached herewith as Exhibit F. On that page, the STAPA 4ZnAl7 paste is noted.
- 21. Concurrently with many of these referenced tests, MCII initiated the preparation of a patent application directed to, among other aspects, the use of various zinc pastes including the 4ZnAl7 paste and the 4ZnSn30 paste in corrosion resistant coating compositions. That application was filed on February 14, 2001 as U.S. provisional patent application 60/268,273.
- 22. MCII periodically conducts "Working Group Meetings" in which various personnel from MCII, NDS, and Dacral meet, generally at MCII's facilities, and discuss numerous projects. On March 7 to March 9, 2001, a Working Group Meeting was held in which the results of various testing of STAPA 4ZnAI7 was

discussed. See pages E-3 and E-4 of Exhibit G. At that same Working Group Meeting, NDS presented its information concerning alloy pastes of zinc and aluminum. 4ZnAl7 is specifically and repeatedly noted. See E-14, E-19, and E-22. It is also noted that "Eckart can provide 5%, 20%, 30% Al" pastes; that "MCII will do industrial trial with 7% Al"; and that "MCII ordered a second batch of 7% Al to test for reproducibility." See E-28. Another reference to this paste is noted on E-34.

- 23. During that same Working Group Meeting noted in paragraph 22, the 4ZnSn30 zinc/tin paste was discussed. See E-2 and E-4 of Exhibit G.
- 24. MCII issued Purchase Order 000610-00 on March 7, 2001 (see Exhibit H) for 1,000 kg of STAPA 4ZnAl7 paste from Eckart.
- 25. The purchase noted in paragraph 23 was confirmed by Frank Passen of Eckart in an email dated March 8, 2001 (Exhibit I).
- 26. MCII issued Purchase Order 000671-00 on March 29, 2001 (see Exhibit J) for another 1,000 kg of STAPA 4ZnAI7 from Eckart.
- 27. MCII issued another Purchase Order from Eckart No. 000957-00 on July 9, 2001 for other STAPA pastes to confirm a verbal phone order of March 19, 2001 (see Exhibit K).
- 28. The origin of the term "STAPA" in association with the pastes at issue is unknown. However, it is believed that upon Dacral requesting and specifying the pastes from Eckart; and Eckart providing to MCII, NDS, and Dacral, the requested (i) zinc/aluminum alloy paste containing 7% aluminum and designated as 4ZnAI7, and (ii) zinc/tin alloy paste containing 30% tin and designated as 4ZnSn30, Eckart incorporated these pastes into their currently existing STAPA line of alloy pastes.
- 29. We further declare that all statements made herein of our own knowledge are true and that all statements made on information and belief are

believed to be true; and further that these statements were made with the knowledge that willful false statements, and the like, so made are punishable by fine or imprisonment, or both, under Section 1001 of title 18 of the United States Code and that such willful false statements may jeopardize the validity of the above-referenced application or any patent issuing thereon.

Terry E. Dorsett

Donald J. Gunde

OCTOBER 8, 2004

Date
N:\MCII\200004\MAT0001578V001.doc

Date

DACRAL S.A.

Regu: USU/OU Vu: A/A (OS Class: France MP/R

17/12/1999

GOMP (F-RENDE REUNION DU 28/11/1999

SOCIETE DÜKAL //SOCIETE+CKARTErancı

Participants:

Sté DORAL

Mr CHAPPEX

Mr STUCKER

Ste ECKART- France

Mr BACOS

Sté DACRAL

MILE SECOUET
MM LELONG
MANETY

MILLET PELLETIER

Copic: M. FOUREZ

### 1) OUALITE DE LA PATE ZINC REF

- Problème de stabilité (évolution du pH)

Constat DACRAL: Depuis mai 1999 jusqu'à mi-novembre 1999, évolution à la hausse du pH. Période plus longue que les années précédentes (juin - septembre 98) (voir document joint).

Autre constat: en une semaine, délai moyen entre le contrôle DORAL et contrôle DACRAL, évolution du pH de 0,2 point. Problème de stabilité qui se répereute sur nos productions. La pâte de Zinc continue à évoluer à l'intérieur de nos produits. Problème d'appariement avec les solutions chromiques chez nos licenciés.

Explication DORAL

Raison atmosphérique: climat humide et doux jusqu'à maintenant

Raison matériel

: malgré l'installation de sécheurs d'air à base d'Alumine ou

Silicagel, dans le circuit production, faible efficacité du système. DORAL prévoit une révision complète de l'installation pendant

l'hiver.

Conclusion: Cibler un pli 24,2

FILE No. 086 08/26 '04 16:55 ID:METAL COATINGS INTL INC. FAX:14402855009

DACRAL S.A.

17/12/1999

### 2) CAPACITE DE PRODUCTION DORAL

→ Actuellement : unité de 2 tonnes par 24 heures soit 600 T/an

→ Prévision : existence d'une unité de capacité identique non encore mise en service.

Nouvelle unité de production : 1<sup>ère</sup> tranche de 800 à 1000 T de plus

→ opérationnel au 16me trimestro 2000

2<sup>nd</sup> tranche qui permettrait de doubler la capacité

actuelle

Rq: L'extrait sec de la pâte de zinc est obtenu uniquement à la presse. Aucun rajout n'est effectué.

### 3) CRITERES SELECTIFS SUR LA QUALITE DE LA PATE DE ZINC

- Influence sur la corrosion
- Influence sur la cohésion
- Fiabilité des pII. Qualité constante de la pâte de zinc
- Avantage et inconvenient de la présence de White Spirit
  - effet bénéfique sur le moussage
  - défavorable pour la cohésion
  - quantité limite 8% dans la pâte de zinc
- extrait sec constant
- qualité des agents de broyage
- qualité des solvants
- critère de prix

Conclusion: Trouver un compromis entre les performances et le coût.

Rq: DACRAL ne souhaite pas utiliser de poudre de zinc dans les productions de DACROMET® pour plusieurs raisons:

- problème de prix
- problème de mise en œuvre risque d'incendie
- /7 stabilité de la poudre de zinc évolution du pH
  - -> éventail plus important de solution chromique.
  - stabilité des composés problème de bullage

### 4) DEVELOPPEMENTS POTENTIELS

- Production de pâtes à partir d'alliage tel que par exemple :
- zinc / étain
- zinc / nickel
- zinc / aluminium

Tout développement est envisageable, il est également possible de produire des unités pilotes de 200 à 300 kg.

Etude demandée: alliage zinc / aluminium (90/10)

DACRAL S.A.

17/12/1999

Ajout d'additif:

But: baisser la température de cuisson des composés (θ < 300° C)

→ possibilité de diminuer le taux de stéarine et de compléter avec un autre additif.

Zinc Noir / Aluminium Noir : (étude R&D DACRAL)
 Echantillon de zinc noir référence UFB : (déjà testé par MCII)
 Echantillon d' Aluminium Noir référence PIRO – MT
 référence PIRO – 5413

Echantillons d'Hydrolac (aluminium à l'eau)

Addition d'un stabilisant pour meilleure tenue à 40° C

Référence W 8 NL → 100% de stabilisant

WH 8NL > 50% de stabilisant

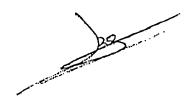
WHH 8 NL → 25% de stabilisant

Echantillon Hydrolux 500 (aluminium) – coût plus élevé mais pouvoir couvrant plus important.

### 5) REMARQUE DORAL

- Logistique NOF Europe Responsable B. SECOUET

  Délai trop court difficulté d'approvisionnement → demander à augmenter le nombre de containers.
- Revoir avec NOF Europe les commandes en tonnelet ou en fûts problème de dédouanement.



14:11 111/83

regu de : +33 1 44293599

Fr. Draes/Hr. Th. Kruse jun. c/o Ewald Dorken AG Wetterstr. 58

58313 Herdecke

Inr Zeichen

Ihre Nachricht Vom

Unser Zeichen

D 91234.Velden, den 10.12.96 Tel.-Nr. (09152)77-572

Dr.FE-za

Teletax (00152)77-551

Sehr geehne Frau Drees, sohr geehner Herr Kruse.

vor niniger Zeit hanen wir über Zn/Al-Legierungspigmente gesprochen und diesbezüglich Versuche vereinbart, Inzwischen steht ein Muster

VP. ZN 624 083

zur Vorfügung, das Sie mit getrennter Post erhalten.

Legierungszusammensetzung: 90 % Zink/10 % Aluminium

Flüchtiger Anteil:

-11%

Teilchengrößenverteilung:

s. Anlago

optische Eigenschaften:

s, Anlage

Für weltere Fragen stehen wir gerne zu ihrer Verfügung.

Wir wünschen ihnen ein frohes Welhnachtsfost und alles Gute im Neuen Jahr und verbielben

mit freundlichen Grüßen

ECKART-WERKE

i. A.

Dr. Andrea Fetz

974 - Eckart Werke VISH report

Home > Tools > Babel Fish Translation > Translated Text ( )

### Babel Fish Translation ®

### In English:

POTENTIAL DEVELOPPEMENTS Production of pastes have d'alliage part such as for example: zinc/etain zinc/nickel zinc/aluminium Any development is possible, it is also possible to produce pilot units of 200 A 300 kg. Study demandee: alloy zinc/aluminum (90/10) d'additif Addition: Drank: to lower the temperature of cooking of compose (0 possibility of decreasing the rate of stearine and of supplementing with another additive. Black Zinc/Black Aluminium: (study R&D DACRAL) black zinc Sample reference UFB: (dega tests by MCII) Sample d'Aluminium Noir reference PIRO - MT; reference PIRO - 5413 Samples d'Hydrolac (aluminium has l'eau) d'un Addition stabilizing for better held has 40 deg C Reference W 8 NL 100% of stabilizing WH 8 NL 50% of stabilizing WHH 8 NL 25% of stabilizing Echantillon Hydrolux 500 (aluminium) - cost more eleve but more important covering capacity.

Search the web with this text

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DEVELOPPEMENTS POTENTIELS

Production de pates a partie d'alliage tel que

par exemple:

zinc / etain

zinc / nickel

zinc / aluminium

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### **FAX TRANSMISSION**

### PAGE 1 OF 7 (INCLUDES COVER LETTER)



### NIPPON DACRO SHAMROCK CO., LTD

296, SHIMOKURATA-CHO, TOTSUKA-KU YOKOHAMA-CITY, KANAGAWA-PRIF. JAPAN, 244

DATE:

February 7, 2000

LOCATION:

METAL COATINGS INTERNATIONAL Inc.

ATTN .:

Don Guhde

C.C.

Mr. T. HIGASHIYAMA, Mr. M NAKAZATO - NDS

FAX#

0042-1-440-2791483

FROM:

KAZUAKI SUGANO (TECHNICAL DEPT.)

SUBJECT

Answer of your fax at February 6.

Dear Don.

Send our formulation and the order of addition. (Attachment 1)

When I made an alloy bath, I mistook an amount of thickener. I added the cellosize at 0.5%. The viscosity was too high. So when I applied bolts, I diluted the bath by de-ionized water and spin speed was high, too.

See the attachment 2 about treating condition and coating weight.

About Alloy GEOMET, how is appearance? Our Alloy GEOMET (only basecoat), white rust is much less than GEOMET 321. The surface of bolts cured at 270, 300℃ was bright, and 330, 360℃ were dull.

Send our latest results of corrosion test (attachment 3-6) and results of second trial. (Attachment 7)

We have a problem that the performance of GEOMET 321 without plus is lower than MCII and DACRAL. The best result of SST was 240 hours. Coating weight was 300 mg/dm². What are important points for corrosion resistance?

Best regards,

Kazuaki Sugano.

Kazuaki Sugano

PLEASE CALL FAX +81-45-871-6281 OR PHONE +81-45-864-4251 IF YOU DO NOT RECEIVE ALL PAGES OR HAVE QUESTIONS.

## GEOMET 321

	ರಾ	20.6	20.6	
	%	50.00	20,00	
GEOMET G1	MoO3 paste	DPG	MoO3	

MoO3 Paste	7.12	41.2
DPG	8.11	47.0
NP4/204.5	7.90	45.7
NP9/208.5	7.90	45.7
Silane A187	14.66	84.9
Al in DPG TOYO	6.70	38.8
Mix for 1 hour with the Conn Itt blade	e Conn Itt blac	je
Eckart dry Zinc	44.31	256.6
Mix for 30minutes with the Conn Itt blade	h the Conn Itt I	blade
Mneral spirit	3.30	19.1
Mix for 30 minutes with the Conn Itt blade	th the Conn Itt	blade
total	100.00	579.0

	413.2	5.9	1.9	421.0
	98.15	1.39	0.46	100.00
<b>GEOMET G2</b>	De-ionized water	boric acid	Ce(NO3)3 crystal	total

1000.0	1.000	
421.0	0.421	GEOMET G2
579.0	0.579	GEOMET G1

1000.0	5.0
1.000	0.50

QP4400H

## **GEOMET Alloy**

### GEOMET G1

<b>D</b>	50.0	14.0	16.0	7.2	85.0	5.2		344.0		521.4	
%	9.59	2.69	3.07	1.38	16.30	1.00	uniform	65.97	Conn Itt blade	100.00	
	DPG	NP4/204.5	NP9/208.5	Niper S10	Silane A187	Aerosol TR70	Mix for 5 minutes to 1	4ZnAl7	Mix 2 hours with the Conn Itt blade	sum	

## **GEOMET G2**

		•
De-ionized water	98.41	376.1
boric acid	1.59	6.1
	100.00	382.2

7 521.4	3 382.2	
. 0.577	0.423	
	•	
T.G1	T G2	
GEOMET G1	<b>GEOMET G2</b>	

903.6

1.000

QP4400H 0.30 2.7

The thicknener is added just after mixing G1 and G2

### Dean Fuller

From:

"Frank Passen" <fpassen@eckartamerica.com>

To:

"Peter Stucker" <stucker@doral.ch>

Cc: Sent: "A Dean Fuller" <purchasing@dacromet.com>

Subject:

MCII

Tuesday, September 05, 2000 11:55 AM

Dear Peter,

Dean asked me to get the complete story on your request for charges regarding the special alloy. We assume that the \$8,600 dollars is the portion of costs to go to MCII, Dacral and NDS. Dean said that since this was a partnership, George Palek would also like to know what were the costs involved for Doral. These costs do not have to be detailed, but George wants to know how the costs are being shared by all.

I have asked Dean to assign the 16 X 50 Kg drums for evaluation. He is also checking on the pH testing solution.

I will let you know as answers become available.

Best regards, Frank

		. •1		
	1	, i.e.		
9924-2	<del></del>			_
GEOMET® FROM		<del> </del>		
Zn/Al ALLOY FLAI	i		41/11 51 455	
ENALALLOT I LA	%	20.250	4 1/4" BLADE - 6 GALLON PLASTIC PAIL	!
DI WATER	18.9	20,350 g	DIOCOLVE	
BORIC ACID	0.6	3846.2	DISSOLVE AS USUAL, COOLED TO 44°F	
DOING AGID	0.8	122.1	ADD A HIV A CANADA	_
OOW Z6040 (45°F	=) 3.0	610.5	ADD & MIX 2 - 3 HOURS	
	7 3.0	010.3	TEMP AFTER 15 MINUTES - 57°F	
			ADD & MIX - LEAVE A PORTION OUT TO USE	
			AS	
DI WATER (51°F)	34.0	6919.0	A RINSE	
NO COOLING DUF	RING THIS PERIC	OD		
NIPAR	0.7	142.5	COMBINE NEXT 5 INGREDIENTS AND ADD AT LOW	
DPG	. 1.2	244.2	SPEED	
<u>IP-4</u>	0.8	162.8		
1P-9	0.8	162.8	·	
OOW Z6040	2.0	407.0		
Batch # 01	1/17		INCREASE SPEED TO 500 RPM, ADD PASTE	
•	* 1	1	AND INCREASED SPEED AS NEEDED -	
W ZINC ALUMINU	M		LOWERED BLADE TO 1/2" FROM BOTTOM -	
ASTE			MIX 3 HRS.	1
TAPA 4 ZnAi 7	32.6	6634.1	@ 600 RPM'S	H (1
WITCH TO A LOV	NI IET ENIOPE	DLADE		7 5
THE TO A LOV	V LIFT FAUCE I	BLAUE		]     0%
•	[		ADDED AND BATH APPEARED TO BECOME 2	# 5 2 H
			PHASE -RAISED BLADE TO MIDDLE OF THE	1 2 3
			BATH FOR 1.5 HRS AND LOWERED TO 1"	1 2 %
	ł		FROM BOTTOM AND CONTINUED TO MIX - ~	1000
	ļ		3 HRS AFTER THICKENING THE PHASING	
			STARTED TO BREAK UP, THE RPM'S WERE REDUCED AND BATH WAS MIXED OVER	
EROSOL	0.4	81.4	NIGHT.	DA BA VIS
2/20/00				1 1 - 1
LIER THROUGH	A 60 MESH SCR	EEN AND T	RANSFER TO THE COLD RPM. MIX FOR 5 - 7 DAYS @ 55°F	£ 2 H
		. ,		1 000
2/27/00				20
EIGHT OF BATH:	17.94kg		RATH @ COMPLETION 470 to 1	280
DAIN.	·····		BATH @ COMPLETION: 17940g / 0.95 = 18884.21g	55
OW Z6040	2.9	547.0	ADD DOW AND MIX 1 LIGHT	
	2.0 (1.0)*		ADD DOW AND MIX 1 HOUR	x x
WATER	0.1* - 0.2	18.9	TRY 0.1% CELLOSIZE IN 1% WATER TO GET 28 -40 SECONDS WITH A # 2 ZAHN	NEL - AFTER BEFORE
	- J J.2	10.0	OLOGRADO WITH A # 2 ZAMN	
	<del></del>			PANEI WT AF
ELLOSIZE			// 3 7 % / -	<b>\$</b> \$\$
ELLOSIZE	TY AFTER THICK	ENING - 24.	19SEC @ 68°F - 42 Zahn	
ELLOSIZE 2/28/00 - VISCOSIT			1020 @ 301	1 2 2 3
ELLOSIZE 2/28/00 - VISCOSIT POKE TO GTD AB	OUT VISCOSITY		19SEC @ 68°F - # 1 Can A	
ELLOSIZE 2/28/00 - VISCOSIT POKE TO GTD AB	NEL - 12	AND HE SA	ID IT WOULD BE FINE.	
PA	NEL - 12	AND HE SA	ID IT WOULD BE FINE.	
ELLOSIZE  2/28/00 - VISCOSIT  POKE TO GTD ABOUT TO GTD AB	NEL - 12	AND HE SA	ID IT WOULD BE FINE.	
ELLOSIZE  2/28/00 - VISCOSIT  POKE TO GTD AB  alflake.xlsSheet1	OUT VISCOSITY  NEL - 12  TAFTER - 13  TBEFORE - 13	AND HE SA 3. 8603	DATE - 12/28/20 BATH - 9924-2 VISC 2419868 6817	
ELLOSIZE  2/28/00 - VISCOSIT  POKE TO GTD ABOUT PART PART PART PART PART PART PART PAR	NEL - 12 TAFTER - 13 TBEFORE - 13	AND HE SA 3. 8603 3.5102	DATE - 12/28/00 BATH - 9924-2 VISC H19866 687 DD BAR - 20	
ELLOSIZE  2/28/00 - VISCOSIT  POKE TO GTD ABOUT PART PART PART PART PART PART PART PAR	OUT VISCOSITY  NEL - 12  TAFTER - 13  TBEFORE - 13	AND HE SA 3. 8603 3.5102	DATE - 12/28/20 BATH - 9924-2 VISC 2419868 6817	
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ELLOSIZE  2/28/00 - VISCOSIT  POKE TO GTD ABOUT PART PART PART PART PART PART PART PAR	NEL - 12 TAFTER - 13 TBEFORE - 13	AND HE SA 3. 8603 3.5102	DATE - 12/28/00 BATH - 9924-2 VISC H19866 687 DD BAR - 20	
ELLOSIZE  2/28/00 - VISCOSIT  POKE TO GTD AB  Ifflake.xlsSheet1  WT  DIF	OUT VISCOSITY  NEL - 12  T AFTER - 13  T BEFORE - 13  FF - 0.3501  WT - 157	3.8603 3.5102	DATE - 12/28/00 BATH - 9924-2 VISC 4/19 40 68°F Cache # 2 DD BAR - 20 AGE - 9 damp	
ELLOSIZE  1/28/00 - VISCOSIT  POKE TO GTD AB  Iflake.xisSheet1  WT  DIF	OUT VISCOSITY  NEL - 12  T AFTER - 13  T BEFORE - 13  FF - 0.3501  WT - 157	AND HE SA 3. 8603 3.5102	DATE - 12/28/00 BATH - 9924-2 VISC 4/19 40 68°F Cache # 2 DD BAR - 20 AGE - 9 damp	3/24/04

## WORKING GROUP MEETING

MCII MARCH 7-9TH, 2001 Chardon, Ohio

EXHIBIT

EXAMELEGALO

### **TABLE OF CONTENTS**

	<u>Page</u>
<ol> <li>GEOMET® From Alloy Flake</li> <li>A. Stapa 4 Zn Al 7</li> <li>B. Zinc/Tin Alloy (Stapa 4 Zn Sn 30)</li> <li>C. Patent Application</li> </ol>	1-2
2. GEOMET® 321 A. High VOC B. Low VOC	3
Low Cost GEOMET® and GEOMET® Alternatives     A. Zinc/Silicate Formulations	3
4. DACROMET 320® LC	4
5. DACROLUB® 10 L and 15 L	4
6. Pre-treatments/Post-treatments	4-5
7. Topcoats	5
8. Aluminum In Mineral Spirits	5
9. Dorken Products	5
10. Zinteck	5



### MARCHARER 2001 METALEGOVILVES INHERVATIONE MORVANGEROUPMEETINE

### 1. GEOMET® FROM ALLOY FLAKE

### A. Stapa 4ZnAl7

The first Zn/Al Alloy GEOMET® bath (ZA12) prepared by Gilbert Lelong was made on June 12, 2000. This was a high VOC bath, prepared using an A/B slurry method. Corrosion resistance was exceptional on the 2+1 with PLUS® L (2184 hours with no red rust). Non-topcoated parts showed first red rust at 144 hours.

The second Zn/Al bath was 9926-42. This was an attempt to make a lower VOC bath, still using an A/B slurry approach. Originally formulated with 2.2% DPG, additional DPG had to be added to make a fluid grind. Parts coated with this bath showed first red rust (2+0) at 240 hours. Parts (2+1) with PLUS® L are still in test at 4128 hours with no red rust (*Figure 1*).

The third Zn/Al bath was 9930-41. This bath was made to be low VOC, and used the standard premix method used for GEOMET®. Parts from this bath (2+0) showed first red rust at 168 hours. Topcoated parts (2+1) with PLUS® L are still in salt spray at 2472 hours. All parts had been cured at 316°C at this point (Figure 2).

The fourth bath (9924-2) was an attempt to produce a low VOC bath with about the same cost as standard GEOMET®. Reports from NDS that exceptional salt spray results were being obtained with 2+0 coatings prompted a cure ladder with 9924-2. The cure ladder on this 1.6% wetter bath failed to show any improvement in salt spray resistance above 316°C (*Figures 3 & 4*).

A portion of bath 9924-2 was removed and was used for a wetter ladder/cure ladder. Up to 7% wetter (total NP4/NP9) was post-added to the bath. Parts coated with this bath are showing a trend of improved salt spray with increasing wetter and increasing cure temperature (*Figures 5 & 6*).



A coating weight ladder was done on the 1.6% wetter bath (9924-2) on bolts. Parts were tested with and without PLUS®, PLUS® L, PLUS® ML, and PLUS® XL (2+1). All the basecoats were cured at 316°C. Some 1+1 variations were included (*Figures 7, 8, & 9*).

Currently, we have prepared a bath duplicating the NDS bath. While it is high in VOC, it will be applied to parts, rotors, etc. to see if the excellent salt spray results can be duplicated (2+0).

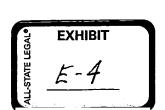
### B. Zinc/Tin Alloy (Stapa 4ZnSn30)

GEOMET® baths made with this alloy replacing the zinc and aluminum produced coatings with excellent tape adhesion on panels and bolts. The optimum cure temperature for these coatings appears to be 232°C. Parts in salt spray have shown no red rust after 288 hours when cured at this temperature (2+0). All the baths were prepared with a similar, low VOC formulation, but with slight wetter variations and manufacturing variations. Initial evaluations showed salt spray resistance of only 72 hours to first red rust (2+0, *Figure 10*). Parts with PLUS® L showed first red rust at 648 hours. A high temperature cure ladder showed no benefit from curing up to 371°C (*Figure 11*). A low temperature cure ladder showed best performance with a cure temperature of 232°C (*Figure 12*). Parts from this evaluation were also coated with PLUS® L (2+1) and are in test.

### C. Patent Application

- 1) Patent Docket #003744 has been filed for applications using alloy flake.
- Support work for this application included preparation of solvent-based GEOMET® formulations with a titanate binder and a mixed silane-titanate binder. Four baths were prepared, two with the zinc/aluminum alloy flake and two with zinc paste/aluminum paste as in GEOMET®. These formulations were based on the original solvent-based, titanate coatings reported at the 1994 International Meeting (RFN-94-1-NC).

These baths were applied to bolts and tested with and without PLUS® L. The alloy flake / titanate / silane coating showed first red rust at 480 hours, compared to the mixed zinc paste/aluminum paste coatings, which showed first red rust at 552 hours. Topcoated bolts are still in test (*Figure 13 & 14*).



Metal Coatings International Inc.
Working Group Meeting
March 7<sup>th</sup> to 9<sup>th</sup>, 2001
Dacral Presentations

### GEOMET® "321": Zn ALLOYS



### I/Zn/Al ALLOY (93/7) FROM ECKART (G. Lg)

### a) Test results

\* Wetting agents : 7%

Silan : 8,5%

Zn ratio (Zr/AL) : 29%

M<sub>o</sub>O<sub>3</sub> : 1-2%

Baking 20' - 70° C + 25' - 300° C

SST (H) on	Without PLUS®	With PLUS® L
<b>bolts</b>	$(31 \text{ g/m}^2)$	$(+ 4 \text{ g/m}^2)$
G 321 réf.	<b>⑤</b> 300-400	⑤ > 1500 H
G 321 (allov)	<b>⑤</b> 430-1000	© 860-2100

\* Wetting agents: 4%
Silan: 8,5%
Zn/Al alloy: 32%

(metals amount <u>=</u> to reference)

 $M_oO_3$  : 2%

Baking  $20' - 70^{\circ} \text{ C} \div 25' - 300^{\circ} \text{ C}$ 

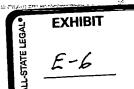
SST (H) on bolts	(25 g/m²)	(+ 5 g/m <sup>2</sup> )
G 321 réf.	<b>⑤</b> 340	> 960 > 1500
G 321 (alloy)	S 720	> 960 > 13.03

Let's note: SST > 840 h on panels (ref. and alloy).

### b) Actions Plan

- At constant Metal amount: study of Zn / Al (alloy) + Al mixing between 100 + 0 → 80 + 20
  - baking temperature: 300° C 330° C 360° C
  - comparison with Zn + Al mixings at the same ratio.
- Alloy study at a lower and higher amount of Aluminium (UM and Eckart Cooperation)
- Influence of : Silan, wetting agents and M<sub>0</sub>O<sub>3</sub> ratios.









### II/ Zn / Sn ALLOY (70/30) FROM ECKART (G. Lg)

### a) Test results

* Wetting agent	: 4%	
* Silan	:8,5%	
* Metal amount	: 32%	
( <u>=</u> to reference)	J	
$M_{o}O_{3}$	: 2%	

SST (H) on	Without	With
bolts	PLUS®	PLUS® L
	$(31 \text{ g/m}^2)$	$(+4 \text{ g/m}^2)$
G 321 réf.	<b>⑤</b> 340	> 960 _3m
·G 321	\$ 340	> 960
(Zn/Sn alloy)		>1700)
Zn/Sn + Al	\$ 625	> 960
= 92 + 8		

Baking:  $20' - 70^{\circ} \text{ C} + 25' - 300^{\circ} \text{ C}$ 

Let's note: - SST > 840 h on panels for all products.

- White rust is higher with Zn/Sn alloy.
- Cohesion is better: 3
- The shade is yellowish / brownish
- Taking tendancy during baking

### b) Actions plan

- at constant metal amount : study of Zn/Sn (alloy) + Al mixing between  $92 \div 8 \to 80 \div 20$ 
  - baking temperature: 300° C 330° C 360° C
  - comparison with Zn + Al + Sn mixing at the same ratio
- Alloy study at a lower amount of Sn (U.M. and Eckart cooperation)
- Influence of: silan, wetting agents and M<sub>0</sub> O<sub>3</sub> ratios



國際企業的國際中央大學學學學學學學院或一個主意中的實際企業上的學術學院的大學學士的學術學學學學學學

**EXHIBIT** 



### GEOMET® "321": Zn-ALLOYS (Pase II)

### III/ ACTIONS PLAN SUMMARY AT DACRAL

- Continuation based on Zn / Al
- Continuation based on Zn/Sn
- Cooperation with UNION MINIERE:

\* Zn /Al: 90/10 and 95/5

\* Zn/Ni: Ni < 1%

\* Zn/Co: Co < 1%

\* Zn/Mn : Mn < 1%

\* Zn/Bi: Bi < 1%

\* Zn / Sn

\* Zn / Mo and Zn / Li : not feasible

- Cooperation with Eckart (flakes):

\* Zn / Al: 95/5 - 80/20 or/and 70/30

\* Zn / Sn: 85/15

\* Zn / some hundred PPM of In or Bi or Ga or Ca...  $\rightarrow$  Zn/In/A1/Bi = 500/50/300 PPM

- Comparison (corrosion) between Zn / Me alloy and General Zn + Me for the different combinations patent Tests with others binders (organic/aqueous phases)

Atomized particles and/or flakes

Let's note: cooperation with "trident Alloys limited" (GB) seems not interesting in that area





**EXHIBIT** 



# GEONETO 321 : PROPERTIES INPROVENTI

## NEXT STEPS

Different Aluminium types (TOYAL - ECKART) have been checked (CM) in 2000: - Aluminium types

SA SA						
		Nature of solvant	% of solvant	% of solvant   Value leafing	Size particle	Covered power
L Daniel Strate Control	Alpate 0231	MS/Toluene	35	75%	7 µm	30000 cm²/g
I	Alpate 1265	MS	36	No	8 µm	
energy and compared to	Alpate 8180	MS/Solvesso 100	18/18	No	7 µm	
eranananan (	Alpate 520	MS	26	70%	5 µm	25000 cm <sup>2</sup> /σ
- L	Chromal 8	DPG	35	70%	13 µm	31000 cm <sup>2</sup> /g
Heritari	Stapa 15	MS	35	65%	8 µm	40000 cm <sup>2</sup> /g
	Stapa 30	MS	35	65%	um 9	55000 cm <sup>2</sup> /g
ALL-STATE LEGAL.	ALL-STATE LEGAL. Hydrolac W 80 NL	MS/water	35	No	21 µm	0/
E		MS/SA/BG/water	10/5/5/15	No	20 µm	

E-9

EXHIBIT

➤ This first screening shows the interest of Alpate 1265 for: aspect – cohesion and SST

The advantage of

For example, one experiment results in SS Resistance values: 1100 h (4,8 → 1250 h)

(Silan: 12% - wetting agents: 6%)

➤ On the other hand, MS in Aluminium might be used

chese results need to be confirmed as a function of baking temperature.

Zn Alloys introduction

Ce No3: introduction for aspect

Mixing of: MoO3/Na2MO4 for SS resistance

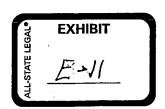
Crosslinking increase: 2 x trifunctionnal silan resins

**EXHIBIT** ALL-STATE LEGAL E-10 子 の様本 こ ちゅ

如果是是一个人,他们就是一个人,他们也是一个人,他们也是一个人,他们也是一个人,他们也是一个人,他们也是一个人,他们也是一个人,他们也是一个人,他们也是一个人, 第一个人,也是一个人,他们也是一个人,他们也是一个人,他们也是一个人,他们也是一个人,他们也是一个人,他们也是一个人,他们也是一个人,他们也是一个人,他们也是一

### Working group meeting at MCII – March 2001 Geomet® and Geomet® II

Nippon Dacro Shamrock Co., LTD. Technical Section



### Contents

- 1. Geomet® formulation
  - 1.1. Geomet® from alloy flakes
  - 1.2. Geomet®321
  - 1.3. Effect of nitrates to Geomet® without water(DPG type)
- 2. Geomet® physical analyses
  - 2.1. SEM observation
  - 2.2. Polarization curves
  - 2.3. Current interrupter
  - 2.4. Electric resistance
  - 2.5. Thermal analyses
  - 2.6. Reology
- 3. Toyota rotor program
- 4. Geomet® line trial
  - 4.1. JPF, wood screw
  - 4.2. Honda, disk rotor
  - 4.3. Mitsubishi, disk rotor
  - 4.4. Honda motor bike, disk rotor
- 5. Top coat on Geomet®
- 6. Quality control
  - 6.1. Curing condition
  - 6.2. Sample confirmation
- 7. Miscellaneous
  - 7.1. Zn/Al flake supply in Japan
  - 7.2. Geomet® on Zn Plating
  - 7.3. Silicate/Zinc formulation
  - 7.4. Molykote® from Dow corning
- 8. Summary

- 1. Geomet® formulation
- 1.1. Geomet® from alloy flakes: Geomet®320 Zn/Al
- Geomet®320 Zn/Al, cure and coating weight ladder

Result: Geomet®320 Zn/Al achieved both salt spray over 1,000 hrs and over CCT-A 20 cycles without plus.

Proper coating condition were curing temperature > 330 °C and coating weight > 195 mg/dm².

### Sample:

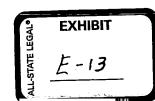
Geomet®320 Zn/Al and Geomet®321 M-10 hex bolt
Dip and spin, 2C2B
100°C-10min -> curing temperature-30min with and without Plus®

### Corrosion test:

♦ Salt spray test

♦ CCT-A

Salt spray, 50°C	17 hrs.
Dry, 70°C	3 hrs.
Soak in salt water, 50°C	2 hrs.
Natural dry, r.t.(20-30°C)	2 hrs.
•	24 hrs

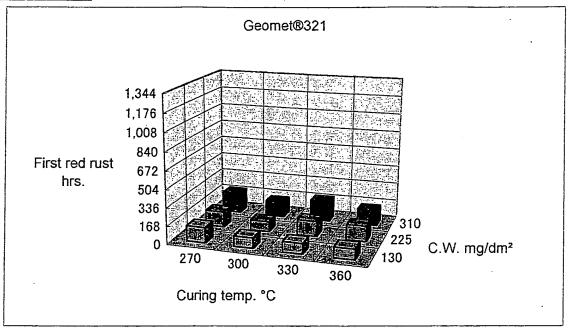


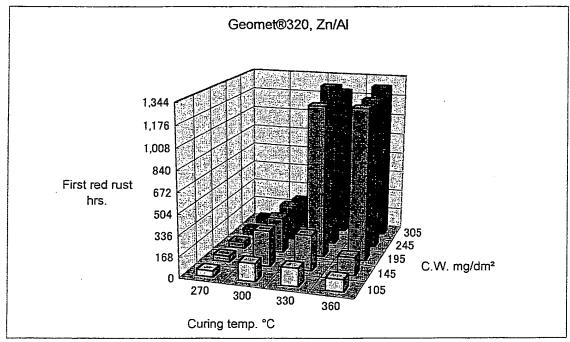
Formulation:

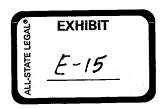
	Geomet®321	Geomet®320 Zn/Al
G1		
Mo paste, 50%	41.2	-
DPG	47.0	60.0
NS204.5	45.7	14.0
NS208.5	45.7	16.0
S-10	-	7.2
A187	84.9	85.0
TR70	-	5.2
Al	38.8	-
Zn, dry	256.6	· <del>-</del>
4ZnAl7	-	344.1
mineral spirit	19.1 ·	-
-	579.0	531.5
<b>G2</b>		
DIW	413.2	375.9
Boric acid	5.9	6.1
Ce(NO3)3	1.9	
	421.0	382.0
total	1000.0	913.5
QP4400	4.0	2.0
pH	7.12	6.89
Zahn#3	30	40
NVM	39%	44%



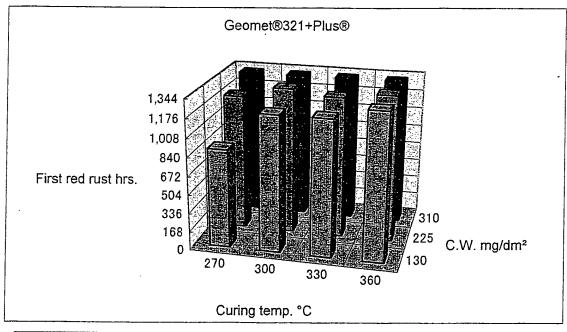
## Without Plus®

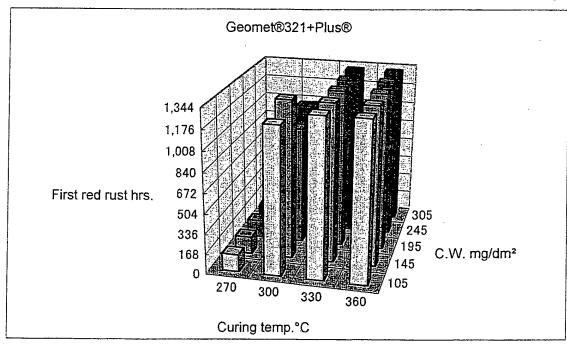




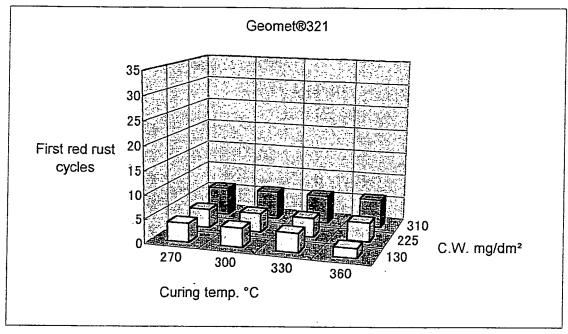


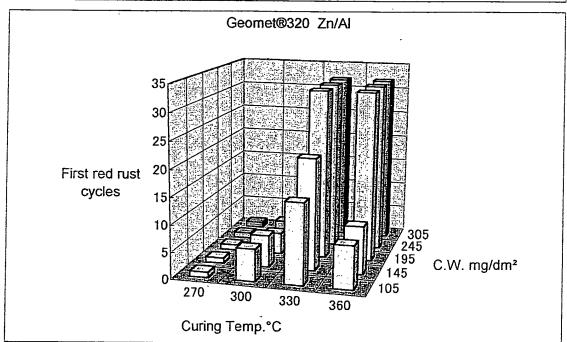
# SST 1,344 hrs. With Plus®

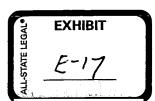




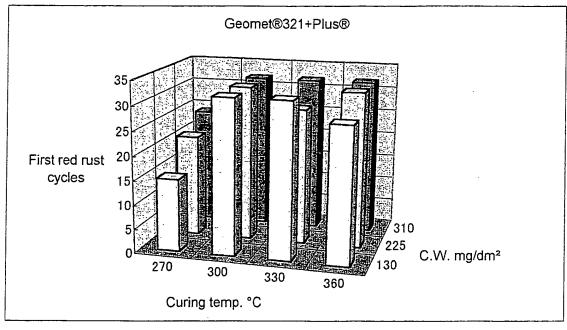
# CCT-A cycles. Without Plus®

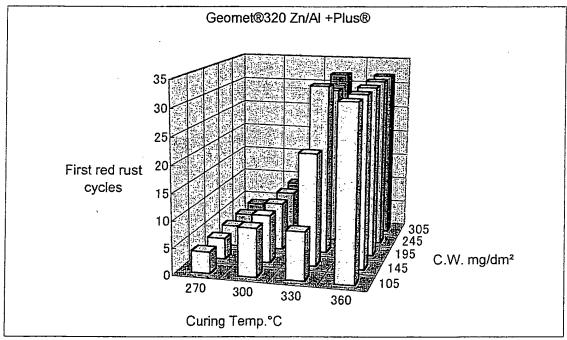


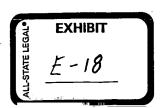




# CCT-A cycles. With Plus®



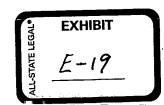




# Investigation of Geomet®320 Zn/Al formulation

# ♦ Binder ladder

		Geomet®	320 Zn/Al	
binder	control	X0.75	X0.5	X0.25
G1			的可能與實施	<b>平立写过一场</b>
DPG	43.3	61.7	80.1	98.6
NS204.5	12.1	<-	<-	<-
NS208.5	13.9	<-	<-	<-
S-10	6.2	<-	<-	<-
A187	73.7	55.3	36.9	18.4
TR70	4.5	<-	<-	<-
4ZnAl7	298.3°	<-	<-	<-
	452.0	452.0	452.0	452.0
<b>G2</b>	多學問題表	對學的數學之外		
DIW	542.7	<-	<-	<-
Boric acid	5.3	<-	<-	<-
	548.0	548.0	548.0	548.0
total	1000.0	1000.0	1000.0	1000.0
QP4400	4.0	<-	<-	<-
рН	6.99	7.01	6.98	6.89
Zahn#3	-28	45	50	27
NVM	(35%	35%	35%	35%
Tape adhesion				
rating	5	4	2	1



## ♦ Binder ladder

SST 72 hrs.

2C2B, 100°C-10min->300°C-30min, 5 - 7 μm

Geomet®320 Zn/Al

control binderX0.75 binderX0.25

red rust on no red rust no red rust scribe bright dull dull dull

## ♦ Cure ladder

SST 120 hrs.

2C2B, 100°C-10min->300°C-30min, 10 μm

	Geomet®	320 Zn/Al	
270 °C	300 °C	330 °C	360 °C_
21.1.14 A	TELET IS	# 7 x 30	WIN A
E. 25 A 25 A 35			
	100 1 100 1 101 1		
		+	
1 7			
A			
7 .		100 B	
Ted Tust off		1	- 1
field and	no red rust,	no red rust,	no red rust,
scribe	bright	bright	dull

STATE LEGAL.

## ♦ Thickness ladder SST 72 hrs.

1C1B, 100°C-10min->330°C-30min

,				
	Ge	omet®320 Z	n/Al	
0.2 µm	1.3 µm	2.4 µm	3.4 µm	7.0 µm
[3030000000000000000000000000000000000				
PART A	01.2.14 A	01.7.14 A	ol. 2.14 A	b ol.214 A to
<b>自然</b> 要证				
				X
			1616	ic h
THE WATER	icib k de	H Tick		
all	all			red rust
surface	surface	red rust	no red	on field
red rust	red rust	on scribe	rust	and

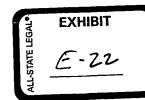
2C2B, 100°C-10min->330°C-30min

,				
	Geo	met®320 2	Zn/Al	
0.5 µm	3.0 µm	3.8 µm	9.3 µm	15.7 μm
OFTH APP	01.2.17 A 414	U.Z.14 A		01.2.14 A
	no red	no red	no red	no red
red rust	rust	rust	rust	rust

## Zn/Al and Zn ratio

# Formulation

	Geomet®320 Zn/Al			
		Zn/A	l:Zn	
_	100:0	66:34	34:64	0:100
G1	<b>经验证</b>			
DPG	43.3	<-	<-	<-
NS204.5	12.1	<-	<-	<-
NS208.5	13.9	<-	<-	<-
S-10	6.2	<-	<-	<-
A187	73.7	<-	<-	<-
TR70	4.5	<-	<-	<-
4ZnAl7	298.3	203.1	108.0	-
Zn, dry	-	95.2	190.2	271.5
	452.0	452.0	451.9	425.2
G2				時期學家的響
DIW	542.7	<-	<-	<-
Boric acid	5.3	<-	<-	<-
	548.0	548.0	548.0	548.0
total	1000.0	1000.0	999.9	973.2
QP4400	4.0	<-	<-	<u> </u>
		· · · · · · · · · · · · · · · · · · ·		
рН	6.97	7.06	7.16	7.36
Zahn#3	32	36	38	29
NVM	35%	36%	37%	35%
Tape				
adhesion	4.5	4.5	4.5	4.5



# Zn/Al and Zn ratio, corrosion resistance

SST 120 hrs. 2C2B, 100°C-10min->300°C-30min, 10 μm

	Zn/A	l:Zn	
100:0	66:34	34:66	0:100
no red rust, bright	no red rust, dull	red rust on field and scribe, dull	no red rust, dull

#### 7. Miscellaneous

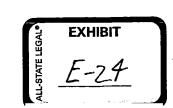
## 7.1. Al/Zn flake in Japan

According to last conference at Dacral in Dec. 2000, we tried to contact with following companies to obtain an alloy powder as a raw material for experimental production of alloy flake in Japan.

### MITSUI MINING & SMELTING CO.,LTD.

At present, we are purchasing zinc flake for Dacrodip® from this company. We had ever obtained some experimental sample of Zn/Al alloy powder from this company about 10 years ago, and we had made an alloy flake for Dacrodip®. This time, we asked sample of the zinc alloy powder processed by gas-atomized method again and started collaboration as follows.

- Mitsui have already thrown away the small Gas-atomized equipment used for last time. They are unable to produce alloy powder by this method.
- Mitsui can produce alloy powder by Water-atomized method. But the crystal structure of the powder becomes harder and more brittle than by Gas-atomized. It's not suitable raw material for flake production.
- ♦ They will investigate other method as soon as possible.
- ♦ In the same time, they will look for the gas-atomized production outside of their company.
- ♦ NDS proposed Al, Co, Ni, Mn, Mg, etc. as an alloy metal with Zn. Mitsui said that it is possible to make alloy powder with Al, Mg. But difficult for Co, Ni etc by Gas-atomized method, because the viscosity of the fused metal becomes higher in the case of such a high melting point of metals.



♦ Recently, Mitsui send us alloy materials by other method as follows.



Zn-0.5%Al alloy material was provided for us from Mitsui. It was not sphere at all, but a ribbon-shaped. The size is very big in comparison with the standard raw zinc powder. (photograph).

This material seems drilling scrap of alloy ingot.

We tried flaking from this material. First of all, flaking wasn't sufficient when made with standard condition. (Shown below). Then milling time was extended and milled for total 40hrs. The milled sample was dried up and evaluated.

Standard Milling Condition for Zn flake in NDS

Equipment

5L-Atriter

Milling medium

3/8 inch steel ball, 10kg

Solvent

Mineral spirit, .2kg

Materials

Zn powder (under 200mesh), 1.5kg

Stearic acid

45g

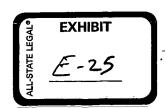
Milling temp.

20 °C

Milling time

20 hrs.

Small amount of flaked powder was obtained after sieve. The evaluation of this flake had started at present.



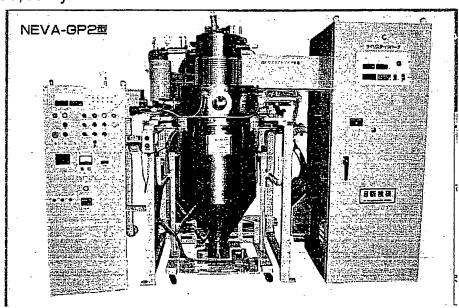
## KOJUNDO CHEMICAL LABORATORY CO.,LTD.

This company is the material manufacturer who handles ceramics, a metal alloy, inorganic material, organic metal compounds and etc, and this company does job shop processing of alloy powder with various method.

The cost is 300,000-400,000yen if we ask 5kg of Zn/Al alloy powder (under  $50\mu m$ ) with Gas-atomized method, and we ordered to produce Zn-4%Al alloy powder.

## NISSIN-GIKEN CO.,LTD

This company manufactures and sells the equipment for material study, like an atomizer and various crystal preparation equipment. The equipment of this company seems to be used even for the above KOJUNDO CHEMICAL company. They can provide small Gas-atomizer (Show below). It can atomize 1-2kg of metal powder at one time and the price was estimated about 22,000,000 yen.





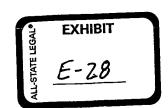
# SUMMARY OF WORKING GROUP MEETING MARCH 7, 8, & 9, 2001 @ MCII - Chardon, OH

## SUMMARY - DAY 1 - ZINC-ALUMINUM ALLOY

	With silicate topcoat, all (NDS, Dacral, MCII) see excellent salt spray performance (more than 1000 hours and up to 4000 hours).
	Without topcoat, NDS formulation had excellent salt spray performance when cured at 330°C or higher. MCII could not duplicate this. Dacral has not yet tested.
٠	MCII found that increased wetter improved SST at 330°C, but still see first red rust at 400 hours even with 5-7% wetter (bolts 2C 2B). DPG may also have an effect and this will be tested.
<b>a</b>	Dacral is testing 321/Alloy, 300°C cure. Without PLUS®: 321 300-400 hours to First Red Rust 321 Alloy 430-1000 hours
	NDS testing alloy with and without MoO <sub>3</sub> , 330°C cure:
	> Mo decreased cohesion
	> Mo may decrease reactivity of Zn/Al too much
	> Zn/Al alloy may be too passive, not enough galvanic protection at the scribe
	> Unusual Result - better corrosion resistance at lower film thickness than at higher
	NDS tested Zn + Zn/Al mixture - As Zn increased, coating appearance more dull, no improvement in corrosion resistance
<b></b>	Patent application submitted February, 2001 – includes GEOMET® and solvent-based titanate and titanate/silane coatings
<b></b>	A table of positive and negative aspects of GEOMET® alloy and GEOMET® 321 was assembled.

# **CONCLUSIONS/ACTION**

		valuate other Zn/Al alloys. Eckart can provide 5%, 20%, 30% Al; also low (<1%) alloys ith Ni; Co, Mn, Bi, and Zn-In/Al/Bi, etc. (used for batteries)
ū	M	ICII will do industrial trial with 7% Al
Ö	M	ICII ordered a second batch of 7% Al to test for reproducibility
		ippon Steel has a Zn/Mg alloy HDG that doesn't need chromating. NDS will try to find ut the Mg content.
	N	DS working with Mitsui and others to get various alloy flakes made
		le should consider making alloys and flake ourselves for testing. Problem is expense of quipment and time to develop expertise.
<u>H</u>	<u>N-</u>	ZINC ALLOY
П		CII Results – poorer corrosion resistance than standard GEOMET®, with and without US®
	>	Excellent adhesion, even at bend in panels
	>	Corrosion resistance is improved at lower temperature (232°C)
	>	Good stability
	NE	OS – galvanic protection at scribe on panels, but not good on bolts
	Da	acral – 321 (with 92% Zn alloy/8% Al flake) better than 321 standard or 321 Zn/Sn
	AC	CTION:
	>	Look at various aluminum additive amounts
	<b>A</b>	Lower Sn content in alloy
	>	Influence of Si, wetter, MoO <sub>3</sub>



#### **GEOMET® 321**

- MCII continues to have trouble with low VOC version, but making progress. Thickener change to Bentone EW, bath without AI, addition of zinc phosphate all help. Cohesion is always poorer.
- □ Dacral evaluated dispersion methods and particle size. Grinding improves smoothness of film, but no effect on corrosion resistance
  - Line trials always had good SST with PLUS® L, but not as good as lab applied without topcoat
  - Finer aluminum paste and increased silane improves aspect, cohesion, SST
- □ NDS Milling experiments with glass beads (3 mm made finer grind than 5 mm) and varying MoO₃/DPG ratio (middle ratio, 33/66, was best).
  - Cerium nitrate decreased corrosion resistance. Higher Mo improved corrosion resistance
  - > 3X wetter improved adhesion and made film brighter, but did not improve corrosion resistance at scribe

## SUMMARY - DAY 2

#### ☐ Physical and Electrochemical Analysis – NDS

- ➤ Polarization curves show MoO<sub>3</sub> moves GEOMET® curve closer to DACROMET® on anodic side, but not on cathodic side.
- > Zn/Al flake reduced corrosion current by suppressing both anodic and cathodic reactions.
- ➤ Corrosion current increased as cure temperature increased, becoming constant above 350°C with GEOMET® 320 Zn/Al alloy.
- Corrosion current increased as binder decreased (GEOMET® 320 Zn/Al alloy) or as zinc flake increased.
- ➤ MoO<sub>3</sub> moved corrosion potential to the inert side, while Co moved it to the active side.
- Current Interrupter method showed no change with GEOMET® 321 cure ladder, while GEOMET® 320 Zn/Al showed high values at high cure temperature.
- GEOMET® 320 Zn/Al was much less reactive than GEOMET® 321.



- > Cure ladders (270° to 330°C) on GEOMET® 321 and GEOMET® 320 Zn/Al showed both improved with higher cure, but GEOMET® 321 was more galvanic on a scribe.
- > Coating weight ladder showed too much GEOMET® 321 Zn/Ai was poor in salt spray.
- ➤ Electrical resistance of GEOMET® 320 Zn/Al is much higher than GEOMET® 321, GEOMET® 320 Zn/Sn, or DACROMET®.
- ➤ Electrical resistance of GEOMET® 320 Zn/Al increases exponentially with film thickness to ~10,000,000 ohms at about 28 microns.
- > Thermogravimetric analysis showed GEOMET® 321 continues to lose weight when cured above 330°C, leveling off at about 450°C.

#### ☐ Toyota Rotor Program – NDS

➤ GEOMET® 321 Zn/Al performed best in salt spray and CCT-C compared to GEOMET® 320, GEOMET® 321, GEOMET® 320 Zn/Sn and GEOMET® 320 Al Rich.

#### ☐ Honda Disc Rotor - NDS

➤ GEOMET® 320 performed better than current black paint in Honda cycle B mode: CCT- H.B. However, GEOMET® is considered too expensive.

#### ☐ Honda Rotor for Motor Bikes - NDS

➤ Appearance of GEOMET® 320 Zn/Sn was appealing to Honda. Shot blasting needed with GEOMET® 320 to pass salt spray (48 hours) test.

### ☐ Togo Hose Clamps - NDS

➤ Togo looking for replacement for DACROMET®. Evaluating GEOMET® 320 Zn/Al and GEOMET® 321, all with PLUS®, PLUS® 55, PLUS® 55 Black and TC 101. At 480 hours, all look OK. DACROMET® and PLUS® control had early red rust. Testing continues.

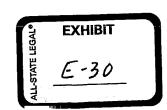
## ☐ Cure Test for GEOMET®

OIM

➤ Soaking cured parts in 1N NaOH for 1 hour appeared to discriminate between parts cured at 270°C, 300°C, and 330°C.

### □ Al/Zn Flake in Japan

- > Mitsui will look into methods for producing alloys.
- > Alloys possible with Mg and Al with zinc.



### ☐ Al/Zn Flake in Japan ...Continued

- ➤ NDS ball milled a sample of Zn 0.5% Al alloy from Mitsui. Evaluations are in progress.
- ➤ Quoted prices for atomizing custom alloys were very high: ~\$2300/5 kg and up.

#### □ Rotor Program - Dacral

➤ GEOMET® 360 (70/30) cured at 340°C performed the same as GEOMET® 360 cured at 250°C + thermal shock (1 hour @ 300°C). >700 hours salt spray and >15 cycles DIN 50017.

#### ☐ DACROMET® LC - NDS, MCII

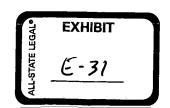
- NDS proposes LC (III), DACROMET® LC with 60% LC additive, 90% CrO<sub>3</sub>, and 50% Al of original LC. Coefficient of friction of this coating is higher than bare steel, and brake noise is same as bare steel. Corrosion resistance was = to DACROMET® 310 KMB @ 2μ and better when LC (III) was at 3μ.
- ➤ NDS tests showed NDS chrome leaching method extracts 2 × the Cr<sup>+6</sup> as ISO 3613.
- ➤ MCII has been producing DACROMET 320® LC (LV) since November, 2000, with no performance problems. MMC is now at 100% LC in both lines.

#### ☐ Molykote D7100 - MCII, NDS, Dacral

- Contains Ti, Si, O, C, P, Zn and Al by EDX.
  - Solvent based system, generally poor crosscut adhesion (tape).
  - ◆ Electrochemically similar to GEOMET® 320.
  - ◆ Topcoat is D708, Black Epoxy with PTFE.

#### □ Dorken – E-Coat – NDS

- Equipment capable of 700 kg/hour. NDS continues to investigate. Adhesion over DACROMET®/GEOMET® is not perfect. Some bare areas noted in recessed heads of bolts.
- MCII test results on Dorken products included in MCII book. Many perform as well as or better than GEOMET® L in salt spray and J2334. None of the test pieces were scribed.



#### ☐ RFN-01-1-PT - Darkening Solution

Demonstration showed ease of use and how depth of color increases with time/temperature. Inhibits white corrosion. Requires no bake or rinsing. Air dry-off or blow-off. Allows topcoating with only one coat of black paint.

## SUMMARY - DAY 3

#### ☐ PLUS® ML - MCII

- DOE showed that varying four major components of PLUS® ML by ±20% had minor effect on average torque (sodium silicate, Polysol M19, Hostaflon 9205, and Polysilk 14).
- > Corrosion resistance exceeded to 500 hours in salt spray on all variations.
- Complete analysis of the data has not been completed.
- Proposed control of PLUS® ML is by % solids rather than viscosity
- Stable, tinted versions of PLUS® ML have been prepared.

#### ☐ PLUS® XL - MCII

Reformulated version is very stable and color versions have been prepared. Torquetension was unaffected by presence of pigment. Salt spray resistance (2+1) over GEOMET® is better than (2+1) with old formula.

#### □ PLUS® L - MCII

Preparation of tinted versions of PLUS® L from a "Base" containing no silicate has been developed. A quantity of PLUS® L "Base" sufficient to make four drums of final product will be done soon at MCII.

#### ☐ Silver Topcoat – MCII

A water-based, silver topcoat has been developed for GEOMET®. Performance 1+1, 1+2, 2+1, and 2+2 is very promising. Air-dry versions can be made with no cross-linker for low-end applications. This topcoat can provide a low-cost, lubricated silver for applications when GEOMET® ML or GEOMET® L are inadequate (wheel cleaner resistance, Kesternich, etc. when barrier resistance is needed).



#### □ DACROLUB 10 & 15

- MCII versions of these products have been showing higher coefficient of friction values than the Dacral formulations. Some of the difference in performance may be due to torque-tension equipment variables.
- Dacral will supply MCII with fresh sample of DACROLUB 10 and 15. MCII will supply DIN 946 test bars to Dacral for comparison.

## ☐ Bulk Spray Application - DACROMET 320®

➤ Work by Dacral on Bulk Spray Application of DACROMET 320® looks promising. Achievement of Grade B coating weights in only 2 coats was done. Grade A is possible in 1 coat. Salt spray was equivalent to Dip/Spin. Kesternich (.2 l) slightly lower than Dip/Spin.

#### □ Black Topcoat for GMW 3359

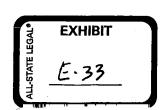
- GEOMET® ML meets all aspects of GMW 3359. GM now is requesting a black version. MCII is looking at:
  - ◆ Black Topcoat, Lubricated
  - ♦ PLUS® ML Black
  - ♦ Modified DACROLUB 15L Black
  - Curtis E-Coat/Seal Coat
- ➤ Most promising are PLUS® ML Black and the lubricated black topcoat. Both can achieve >500 hours salt spray. Magni has no successful candidates at the present time.

#### ☐ GEOMET® Over Zinc Plating

- NDS work on wood screws with GEOMET® over zinc plating showed:
  - ♦ Best performance when cured at 250°C.
  - ♦ High PLUS® coating weight needed for best performance (5 to 6 g/m²).

## □ Polyanaline (Conductive Polymer)

Polyanaline from GeoTech was discussed. This is polyanaline grafted to lignin to make it dispersible. They are commercializing the dispersion with aluminum flake called "Catize". This is used to provide conductivity to cross-linked systems. Lab quantities of "Lignin Pani" grafted material as dispersions are available for \$0.70 to \$3.06 per gram. Catize costs \$0.12 per gram.



#### □ DACROKOTE® Silver

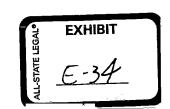
- Dacral presented a paper on DACROKOTE® Silver, topcoat for GEOMET® or DACROMET® with acid resistance.
- > This cross-linked polyester/aluminum system improves 0.2 I Kesternich and salt spray and has excellent adhesion.
- ➤ It reduces leachable Cr<sup>+6</sup> by ISO 3613.
- It can be modified with PTFE or Polysilk for specified torque-tension requirements.

#### ☐ GEOMET® 321 Line Trials

Dacral presented paper on GEOMET® 321 line trials. A topcoat is still required to obtain ≥500 hour salt spray. Performance with PLUS® L was typically >1000 hours. Without topcoat, less than 500 hours. Work continues on improving consistency of results with no topcoat.

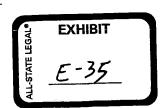
## **ACTION PLANS**

- 1) Zn/Al Alloy Flake
  - a) Increased DPG MCII evaluating NDS formula
  - b) Increased Wetter in low VOC bath MCII
  - c) Use in 321 cured >300°C Dacral
  - d) Dacral/MCII ordered 600 kg of Zn/Al (5% Al) 200kg for each
  - e) Dacral/MCII ordered 600 kg of Zn/Al (20% Al) 200 kg for each
  - f) Investigate in-house dust/flake alloy production MCII/NDS/Dacral
  - g) Order (1 ton) new batch of Zn/Al alloy MCII/Dacral (STAPA 4ZnAl7)
  - h) Trial at MMC with 7% Aluminum alloy GEOMET® MCII
- 2) Other alloys Zinc with Ni, Co, In, Bi, Mn, etc. (Dacral ordered 500 kg) Zn/Sn, Zn/Mg, Others MCII/NDS/Dacral
- 3) Zn/Sn alloy a) combinations with Zn flake, Al flake, etc. MCII
  - b) MCII/Dacral ordered 500 kg Zn/Sn (85/15)



- Continue electrochemical testing at NDS

   (and TGA testing)
   (and EDX for Carbon/Cure ladder)
- 5) Secrecy/Exclusivity Agreement with Eckart will be pursued MCII/Dacral/NDS
- 6) GEOMET® 321
  - a) Improve adhesion with no topcoat MCII/Dacral
  - b) Improve film appearance Dacral
  - c) Low VOC GEOMET® 321 MCII
  - d) Improved corrosion resistance/no topcoat NDS/Dacral/MCII
- 7) DACROMET 320® LC (LV)
  - a) 1st Coat Cure Ladder at MMC MCII
  - b) Continue to monitor performance MCII
- 8) DACROMET® LC (III)
  - a) Continue evaluations NDS
  - b) Low VOC version NDS
- 9) Molykote D7100
  - a) Analyze: Zn/Al ratio MCII
     Si/Ti ratio MCII
  - b) EDX on Topcoat MCII
- 10) Dorken E-Coat
  - a) Purchase by Nagoya Dacro NDS
  - b) Continue discussion with Dorken MCII
- 11) Post-Treatment (NH<sub>4</sub>Cl/NH<sub>4</sub>MoO<sub>3</sub>)
  - a) Field trial at MMC or Apex MCII



## 11) MCII Rotor Programs

- a) Continue evaluation of GEOMET® formulations
- b) Continue development of Zn/Silicate formulations
- c) Low-cost replacements for TRW air-dry black



		DACRAL	NDS	NOI
GEOMET® Alloy Zn/Al	POSITIVE	Corrosion Resistance with PLUS® Ease of Production Functionality (Lower Coating Weight)	High Corrosion Resistance Heat Resistance Easy to Produce Functionality (Lower Coating Weight) Good Adhesion	Ease of Production Bath Stability Good Adhesion Low VOC – Easy to Make Functionality (Lower Coating Weight) High Corrosion Resistance Without PLUS@? High Corrosion Resistance
	NEGATIVE	Cost Patentability?	Cost? Less Sacrificial in Damaged Area Higher Cure: 330°C Patentability?	Cost? 330°C Cure Patentability? One Source Only (Eckart) Reproducibility Unknown
GEOMET® 321	POSITIVE	Bath Stability Improved Corrosion Resistance With and Without Topcoat	Corrosion Resistance With Topcoat	Corrosion Resistance With Topcoat
	NEGATIVE	Cohesion – Adhesion – No Topcoat Film Appearance	Corrosion Resistance – No Topcoat Can Only Use QP4400 in 321	<ul> <li>Making a Low VOC System</li> <li>Cost</li> <li>Corrosion Resistance With No Topcoat</li> <li>Mo Restriction</li> <li>Poor Adhesion</li> <li>Bath Stability</li> </ul>

EXHIBIT

E-37

# DACRAL S.A.

# IC MILLENIUM

Yokohama - 2 - 6 October 2000

Technical session

EXHIRIT

E-38

## B. GEOMET®

B1. GEOMET® 360 (GEOMET® D)

We started to produce GEOMET<sup>®</sup> 360 (which is also the new GEOMET<sup>®</sup> D) in August 99. Today we have produced 60 batches. The batch size has been increased from 1 to 2 tons. When we produced the first batches, we faced a grain problem. The origin was the Cellosize. We switched to Rheolate 255, but we found than the viscosity control was unpredictable. Now we use a Cellosize US quality. We have no more problem. We also have noticed during a certain period a loss of cohesion.

We did not determine the cause.

At our licensee, we had some clogging problem at KAISER which are connected to the spray circulating of their new line. We also had a problem of gazing with one container at AUDI. This container has been stored a too long time outside the 20°C. room. We have tried to find a solution for stopping the gazing. Calcium nitrate has some effect but we did not find a complete solution.

## B2. GEOMET® 320 / GEOMET® 321

In July we decided the formulation which will be offered to our customers for fasteners: it will be GEOMET® 321.

GEOMET<sup>®</sup> 321 contains some molybdenum oxide, which increases the corrosion resistance when the film is not top coated with Plus (480 hrs SST). The initial drawbacks were: poorer film cohesion and poorer film aspect. By adjusting the MoO<sub>3</sub> incorporation process we succeed to improve the film appearance and we found also some leads to solve the cohesion problem.

B3. Spray application of GEOMET® D improvements.

GEOMET® has a tendency to settle in the circulating of the spray equipments. This create some clogging problems, and irregularity in the application. This occurs only when the circulating does not go to the head of the spray nozzles.; We have tried many thickener to solve this problem. KELZAN AR reduces the settling and improves the situation. Stability of the product has to be checked.

B4. Industrial trial with GEOMET® 321 and GEOMET® 360.

We made an industrial trial in FOSFANTARTIGLIO in May 2000.

Corrosion results were:

GEOMET® 360	30 g	168 hrs	
GEOMET® 360 L	30 g + 4	600 – 650 hrs	
GEOMET® 321	30 g + 4 + Dacrolub	500 hrs	
GEOMET® 321 M	30 g + 4	> 1000 hrs	

A second trial is presently done at LEIST.

EXHIBIT

E-39

## ↑ GEOMET® II

In the frame of the GEOMET® II project, we have tried to improve GEOMET® 321. Addition of several metal salts such as Co, Ni, Ce, Mn, Mg have been studied. some of them can improve the corrosion resistance and the film appearance; the most promising is cobalt chloride, but the stability of the bath must be checked. Replacement of DPG by TEG allows to improve the cohesion but the stability of the bath is poor.

Addition of organic resins has also been tried. Corrosion resistance was downgraded. We also found that the replacement of Chromal VIII (DPG) by STAPA VIII improves the cohesion of GEOMET® 321. Bath stability is currently being checked. We tried to introduce other silanes or titanate but found no performance improvement.

Using Zn/Al alloy allow to reduce white rusting and also final salt spray resistance.

#### D. GEOBLACK™

We have tested DELTASEAL GZ as a black top coat for GEOMET® in the following system:

GEOMET® 321 2C + DELTASEAL 1C

GEOMET® 321 2C + DELTASEAL 2C

These 2 systems resists 700 - 800 hrs in salt spray. This is much better than with

DACROKOTE 450. The system GEOMET® 321 + PLUS + DELTASEAL 1C failed rapidly.

E. Complementary products : PLUS L; DACROLUB®.

STUDY 00 189: Behaviour of the following systems to 60 tightening cycles: DACROMET® 500, GEOMET® 321 + PLUS® M and GEOMET® 321 + PLUS® L

## **OBJECTIVE**

In studies 00 127 and 00 180, we compared the influence of 60 tightening cycles on the performance of the assembly (coefficient of friction and tension). The bolt and nut were coated and the washer was DACROTIZED and supplied to us by Renault Industrial Vehicles. We studied the following coatings:

- DACROMET® 500
- DACROMET® 500 + PLUS® L or PLUS® M
- DACROMET® 500 + DACROLUB® 10 L.

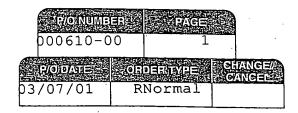
In order to reassure FORD UK in the choice of GEOMET® M / L, in this study we looked at the influence of 60 tightening cycles on the performance of the assembly (coefficient of friction and tension). In this instance, the bolt is coated, the nut is uncoated and the same washer as above was used. We studied the following coatings:

- DACROMET® 500
- GEOMET® 321 + PLUS® L or PLUS® M

## RESULTS AND CONCLUSIONS

The stability after 60 tightening cycles of the systems GEOMET® 321 + PLUS® M or PLUS® L is superior to that of DACROMET® 500.





ORDERED FROM

> Eckart America L.P. P.O. Box 747

Painesville OH 44077

SHIP TO

> METAL COATINGS INTERNATIONAL, INC. 345 INDUSTRIAL PARKWAY

CHARDON OH 44024-1083

BUYER	TERMS	ACKNOW CONFIRM	FOB	SHIP VIA	COL/PPD
A.D. FULLER	NET 30 DAYS	No No	blank	BEST WAY	
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1,000.00 KG STAPA 4 ZN AL / Paste, Zinc/Aluminum

Ext Price =

8,390.00

COMMENTS

This order confirms a.m. phone conversation of 03/07/01 with Frank.

MAYSCUA Marion, Ohio 43302

ORDERED BY



Eckart America L.P. 830 East Erie Street P.O. Box 747 Painesville, Ohio 44077-0747



(440) 354-0400 <del>(440)</del> 354-6224 Fax (800) 556-1111 Toll Free

INVOICE NO.

CUSTOMER NO. 70948

SOLD TO: METAL COATINGS INTERNATIONAL 275 INDUSTRIAL PARKWAY
CHARDON, OH 4400 CE VED USA JUN 2 6 2001

SHIP TO: METAL COATINGS INTERNATIONAL 275 INDUSTRIAL PARKWAY CHARDON, OH 44024 USA

METAL COATINGS INTERNATIONAL

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- west wind I	P.O. Box 7	1369, Cleveland, OH	44191-0569					

#### Dean Fuller

From: "Frank Passen" < fpassen@eckartamerica.com>

To: "Alain Chappex" <chappex@doral.ch>

Cc: "Shauna Maughan" <smaughan@eckartamerica.com>; "A Dean Fuller"

<purchasing@dacromet.com>

Sent: Thursday, March 08, 2001 10:11 AM

Subject: Stapa 4 ZnAl7 for MCII

Good day Alain,

We received today from Dean Fuller his PO#610 for 1,000 Kg of the alloy in 50 Kg steel drums.

Shauna will forward you the paperwork.

He ordered this size on the recommendation of Peter Stucker as this should give MCII a feeling for the

typical large batch.

Once made, these 20 drums should be included in the next available container going to Chardon. Please color these drums differently so they can be distinguished easily from the ZN699. A color band on the side near the top should be adequate.

Thanks and best regards, Frank

# PURCHASE ORDER



Metal Coatings International Inc. 275 Industrial Parkway Chardon, OH 44024-1083 PH: 440.285.2231 FAX: 440.285.5009 www.dacromet.com

	P/O NUMB	ER	PAGE		
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ORDERED FROM

1

Eckart America L.P. P.O. Box 747

Painesville OH 44077

SHIP

METAL COATINGS INTERNATIONAL, INC. 345 INDUSTRIAL PARKWAY

CHARDON OH 44024-1083

	BUYER		TERMS	ACKNOW- LEDGE	CONFIRM	FOB	SHIP VIA		CO	L/PPD
A.D.	FULLER	NET 3	0 DAYS	No	No	Destination	Ocean/Sea/G	rnd		
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1,000.00 KG ZNAL7

Stapa 4, Zinc/Alumimum Paste

8.39 06/01/01

Ext Price = 8,390.00

COMMENTS

This order confirms a.m. phone conversation of 03/29/01 with Frank P.

ORDERED BY Cantuller

## PURCHASE ORDER



Metal Coatings International Inc. 275 Industrial Parkway Chardon, OH 44024-1083 PH: 440.285.2231 FAX: 440.285.5009 www.dacromet.com

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ORDERED FROM

Eckart America L.P. P.O. Box 747

Painesville OH 44077

SHIP TO

METAL COATINGS INTERNATIONAL, INC. 345 INDUSTRIAL PARKWAY

CHARDON OH 44024-1083

	BUYER		TERMS	ACKNOW- LEDGE	CONFIRM	FOB		SHIP VIA		COL/PPD
A.D.	FULLER	NET 3	0 DAYS	No	No	Destination	,	Ocean/Sea/G	rnd	
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2 .	200.0	0 KG	ZN685			te, #ZN684	Ext	Price = 9.95	06/01	1,990.00 L/01
2	250 0	0 80	Stapa #4, ZN683	ZnAl2	0 Pa	ste, #ZN685	Ext	Price = 12.14		1,990.00 1/01
3	250.0	O NG		ZnSn1	.5 Pa	aste, #ZN683	Ext	Price =		3,035.00

COMMENTS

This order confirms a.m. phone conversation of 03/19/01 with Frank.

ORDERED BY DOWN TILL

PURCHASE REQUISITION		,
∠75 INDUSTRIAL PARKWAY CHARDON, OHIO 44024  MICHIGAN METAL COATINGS, CO. 2871 RESEARCH DRIVE ROCHESTER HILLS, MI 48309	REQUISITION 1531	13
METAL COATINGS INT'L. INC. 345 INDUSTRIAL PARKWAY CHARDON, OHIO 44024  GEORGIA METAL COATINGS, CO. 3033 ADRIATIC CT. NORCROSS, GA 30071	957	7
VENDOR SIKART AMORICA C.P. SUGGESTED VENDOR (OPTIONAL)		
P.O. Box 747 PAINESULLE, OH		,
PURCHASE ORDER DATE SHIP DATE REQUISITION DATE		
F.O.B. SHIP VIA REQUIRED DATE	19-01	
OCEAN/SEA  CASH TERMS   <del>, 0, </del>		
ITEM QUANTITY UNIT DESCRIPTION PLEASE PRINT	PRICE DOLLARS CE	NTS UNIT
1 GOOKG STAPA#4, ALZA ZNAL 5 PASTE	8 9	75 Kg
#ZN684		- 1 1
2 500 Kg STAPAH4, ZNAL 20 PASTE	\$ 9.19	75 Kg
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